SIMS CHARACTERIZATION OF NITROGEN DOPING OF LCLS-II-HE PRODUCTION CAVITIES

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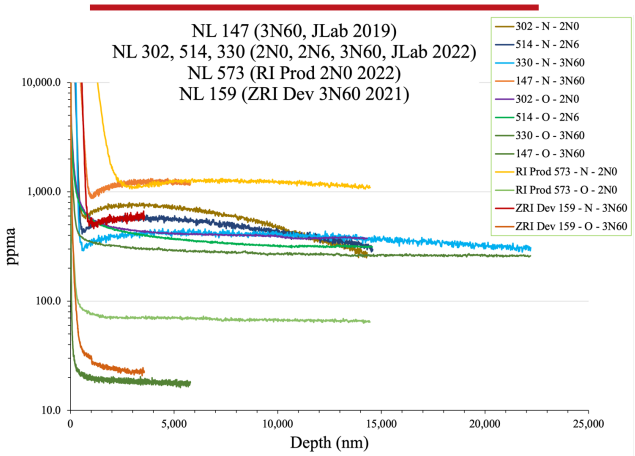
MOPMB006

SRF 2023 GRAND RAPIDS

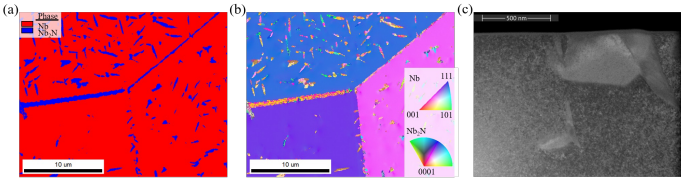
Abstract

The thermal diffusion of nitrogen into the surface of niobium has been shown to yield superior low-loss SRF performance. An effective solution was identified and promptly employed in the production of cryomodules for LCLS-II. With added experience and R&D, a modified process was chosen for use in the upgrade for LCLS-II-HE. Largely motivated by this circumstance, supporting research has significantly refined the technique for making calibrated secondary ion mass spectrometry (SIMS) measurements of the N concentration depth profiles produced by production processes. Standardized reference samples were included with four HE production cavities in their N-doping furnace runs. We report the calibrated dynamic SIMS depth profiles of N, C, and O for these samples, together with the cryogenic acceptance test performance of the associated cavities. Interpretation and comparison with similar samples acquired in other furnaces highlights the importance of intentional process quality control of furnace conditions.

- Four standardized high-surface-quality Nb samples were prepared with ~5 nm roughness
- Included these samples in the production N-doping furnace runs of four cavities for LCLS-II-HE at RI Research Instruments GmbH.
  - Vacuum furnace 800 °C 3 hrs + 2 min exposure to N_2 @ ~33 mbar
- Using refined secondary ion mass spectrometry technique
  - Referencing similarly prepared samples ion-implanted with N, O, and C
  - Cameca 7f dynamic SIMS system at the Virginia Tech NanoCharacterization and Fabrication Laboratory
  - Sputtered with Cs^+ beam
- Acquired depth profile of N, O, and C atomic concentration in Nb matrix via analysis of secondary ions
  - Analysis of three individual Nb grains on each sample
- Excellent reproducibility
- Cavity production process consistency check looks good.
- Vacuum conditions influence N uptake and diffusion.



Variation of N-diffusion characteristic correlates with presence of other species (O & C), compromising process control.



(a) Phase map and (b) orientation map from N doped sample NL 573 showing the formation of Nb nitrides within grains and along grain boundaries. (c) Scanning transmission electron micrograph of N doped Nb sample NL 159 showing the formation of sharp Nb nitrides that intrude into the surface.

Acknowledgements
This material is based on the work of Jefferson Science Associates, supported by the U.S. Department of Energy, Office of Science, Office of Nuclear Physics Early Career Award to A. Palczewski under contract DE-AC05-06OR23177, with supplemental support from Office of Basic Energy Sciences via the LCLS-II-HE R&D program and project funds. J. Angle's support was from the Office of High Energy Physics, under grant DE-SC-0014475 to Virginia Tech.

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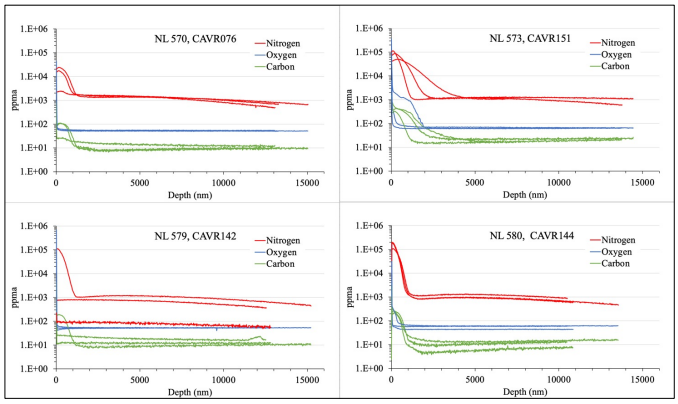

Figure 1: Concentration profiles determined by SIMS of N, O, and C in single surface Nb grains of samples included in production N-doping heat treatments for four cavities for the LCLS-II-HE Project.

Several significant features:

- At the intended surface removal depth of 5 µm for LCLS-II-HE cavities, the N atomic concentrations are all quite consistently ~1000 ppma (0.1%), with one odd exception.
- The depth of the surface nitrides varies significantly among the sampled grains.
- Two grains on sample NL 579 show an absence of any surface nitride signature.
- Indicated surface C concentration structure correlates with that of N, which might be due to higher secondary ion yield from a nitride matrix compared with that of Nb.
- The O and C concentrations beneath the "disturbed" nitride layer are consistent with nominal starting material specifications, indicating insignificant uptake from the furnace vacuum conditions during "doping".
- The N diffusion profiles appear deeper than in previously reported conditions (with higher O and C present), presenting weak sensitivity to modest variations in local removal by electropolishing of the cavities.

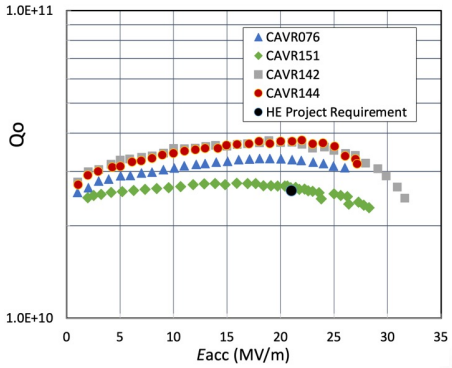

Figure 2: Acceptance test performance for the four HE production cavities with N-doping samples.

The cavity CAVR151 had a higher Q0 prior to some quench processing which very likely induced some transient flux trapping, adding about 1 nΩ additional surface resistance for this test.

Summary

- SIMS analytical methods for characterizing the surface of niobium have advanced in recent years.
- This technique has been applied to assess reproducibility of the thermal loading of the surface of the LCLS-II-HE cavities with nitrogen.
- The production processes used for the "doping" of the surface of LCLS-II-HE cavities with nitrogen appears stable and contributes to cavity performance that exceeds project requirements.
- We note that reproducibility of the actual nitrogen concentration profile is dependent on high quality furnace conditions. Also, grain-to-grain orientation variability may exist that gets integrated into the net whole-cavity performance.

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